

The invention relates to a method for loading a fibrous stock suspension which contains cellulose fibers with calcium carbonate.

When a fibrous stock suspension is processed according to the fiber loading technology calcium carbonate is precipitated. This process has been described already, for example in DE 101 13 998 A1.

It is the objective of the current invention to create additional methods for the production of a fibrous stock which is loaded with calcium carbonate.

In accordance with the current invention this objective is met by a method comprising the following process steps:

- Adding calcium hydroxide in liquid or dry form, or calcium oxide into the fibrous stock suspension,
- Adding of gaseous carbon dioxide into the fibrous stock suspension,
- Precipitation of calcium carbonate through the carbon dioxide and
- Refining of the fibrous stock suspension during the loading process and washing of the fibrous stock suspension after the crystallizing process and/or the refining process and/or during the refining process and/or after the refining process.

Alternatively, this objective is met by a method comprising the following process steps:

- Adding calcium hydroxide in liquid or dry form, or calcium oxide into the fibrous stock suspension,
- Adding of gaseous carbon dioxide into the fibrous stock suspension, and

- Precipitation of calcium carbonate through the carbon dioxide,
- Washing of the fibrous stock suspension prior to feeding the fibrous stock suspension into a headbox chest that is located downstream in flow direction of the fibrous stock suspension and/or into a machine for further processing of the fibrous stock suspension. The fiber loading technology may be applied before or after the refining process, depending upon specific requirements upon the end product.

The current invention describes a method for the production of fiber loaded precipitated calcium carbonate (FLPCC), especially for the production of chemical pulp or utilization of chemical pulp in paper production. The fiber raw material that is to be loaded may – for example – be produced from recycled paper, DIP (deinked paper), secondary fiber stock, bleached or unbleached pulp, mechanical pulp of any type, bleached or unbleached sulfate pulp, broke, linen, cotton, and/or hemp fibers (predominantly used for cigarette paper) and/or any other paper raw material that can be utilized on a paper machine.

The inventive method can be utilized irrespective of whether or not the end product contains a filler that was produced by a precipitation process in a batch reactor or by a refining process (GCC = ground calcium carbonate), or whether talcum, silicon, titanium dioxide ( $\text{TiO}_2$ ), etc. are used.

In accordance with the FLPCC process described below the filler material utilized in other production processes is replaced with the filler material produced according to the fiber loading process technology. The range of application for the filler produced with the fiber loading process technology extends to the production of paper and to applications for all paper grades, including packaging papers that have a filler content

of between 1 and 60% and /or a white liner having a filler content of between 1 and 60%.

The range of application of the current invention is not limited to utilization of these fillers in paper producing processes; the invention can be applied in any paper producing process or related process, including the production of chemical pulp. If a fibrous stock suspension is treated with the fiber loading technology during paper production, a completely new product results which offers new and improved characteristics compared to currently known products on the market. The process described below allows for precipitation of the filler (calcium carbonate) - which is attached evenly distributed exclusively in and on the fiber stock, especially the paper fiber - directly during stock preparation in a paper mill.

A combination or an individual application of the inventive design variations described below results in that only fibrous stock which is loaded with precipitated calcium carbonate is produced, whereby the calcium carbonate is attached on or in the fibers, or embedded in them; this prevents the formation of loose precipitated calcium carbonate (PCC): An additional wash cycle after the refining process and/or during the refining process and/or after the crystallization process in a crystallizer and/or prior to the headbox chest or prior to entry into the paper machine or by returning the press filtrate to a header tank or another storage arrangement on the infeed side provides that a constant calcium hydroxide content is adjusted or regulated in the infeed system of the fiber loading apparatus. The calcium hydroxide can be added directly in a fiber stock pulper. The press filtrate can be returned into the stock pulper system. Calcium hydroxide that is not converted into calcium carbonate or which does not attach to the fibers is again returned to the preceding processes.

Only that filler which is not attached on or in the fibers, in other words loose precipitated calcium carbonate is washed out. The fibers themselves which are provided with filler on the inside and the outside do not lose said filler through the wash process and the recirculation of the press filtrate, so that the positive effects of the fiber loading process are maintained.

In addition to the design forms of the current invention which are described in more detail below, we also refer you to the design examples which are described in further detail in US 6 413 365, DE 101 07 448 A1 and DE 101 13 998 A1 with which the inventive method can also be implemented.

The invention especially comprises a method according to which the fibrous stock suspension is fed into a press arrangement intended to squeeze out a filtrate. Subsequently, the filtrate is directed back, at least partially, into a supply-side arrangement for pulping of the fibrous stock suspension, specifically into a reservoir, for example a header tank. The calcium hydroxide is added at least partially in the arrangement for pulping of the fiber stock. In the complete pulper system, specifically in the arrangement for pulping of the fibrous stock, a pH value of between 7 and 12, especially between 8 and 12 is maintained.

In accordance with the current invention aqueous fibrous stock material, especially aqueous paper stock having a consistency of 0.1 to 20%, preferably between 2 and 8 % is used as primary raw material.

Calcium hydroxide in aqueous or in dry form, or calcium oxide are mixed into the aqueous paper fiber stock in a range of between 0.01 and 60% of the existing solids content. A static mixer, a header tank or a pulper system are utilized for the mixing process; a pH value in the range of between 7 and 12, preferably between 9 and 12 is

applied. The reactivity of the calcium hydroxide is between 0.01 and 180 seconds, preferably between 0.05 and 60 seconds. Dilution water is mixed in according to predetermined parameters, to thereby produce an aqueous primary raw material.

Carbon dioxide is added into the moist paper stock suspension according to the reaction parameters. Calcium carbonate precipitates here in the carbon dioxide atmosphere.

At the same time a refining energy in the range between 0.1 and 300 kWh/ton dry paper pulp is applied. Compared to conventional processes for the production of a fibrous stock suspension, the current invention provides energy efficient attainment of a higher level of freeness; according to the current invention as much as 50% of refining energy can be saved. This affects especially all paper grades which undergo a refining process during their production and particularly those that have a high or very high freeness value, for example FL-cigarette papers (FL = Fiber Loading), FL B&P papers, FL kraft sack papers and FL filter papers. With these papers which do not require fillers, loose filler which is not deposited on or in the fibers can be removed prior to feeding the fibrous stock suspension into the headbox chest or prior to entry into the paper machine. The fibers themselves however, are loaded inside and outside with filler, so that the positive effects of the fiber loading technology are maintained.

The high mechanical strengths in the end product which are achieved through the high freeness value positively affect the production of all paper grades, especially FL cigarette papers, FL B&P papers, FL sack papers and FL filter papers since, due to process based mechanical loads in the various section of the paper machine, such as the press section, the dryer section or in the section where the web is wound, the produced intermediate product and the end product which is to be produced bear a high mechanical load due to utilization of winders, rewinders and converting machinery. Great mechanical stresses occur especially on cigarette paper during its production. These are often caused by

utilization of winders during the production of cigarette paper and by the low basis weight.

The inventive pre-treatment of the fibrous stock suspension also creates the provision for improved drying, thereby increasing the efficiency level in the production of all paper grades. Residual moisture contents in the range of 1 to 20% are advantageous.

An additional advantage of the current invention is that greater brightness and/or higher optical values with around 15 or more lightness points are achieved on all grades of paper, cardboard or in various cardboard applications including the white liner on a cardboard layer.

The energy supply during the refining process, specifically the heat volume and the resulting warming effect are controlled. Crystals in various forms can be produced, according to the control.

In an additional embodiment of the method the current invention provides that a static mixer, a refiner, a disperger and/or a fluffer FLPCC reactor are utilized as a reactor, whereby the fibrous stock content, especially the paper content is between 0.01 and 15% in the instance of a static mixer; at between 2 and 40% in the instance of a refiner and a disperger, especially between 2 and 8% for LC refining and between 20 and 35% for HC-refining and between 15 and 60% in the instance of a fluffer-FLPCC-reactor.

The current invention also relates to a method according to which an expenditure of energy of between 0.3 and 8 kWh/t, especially between 0.5 and 4 kWh/t is used for the precipitation reaction, especially if no refining process is utilized.

The process temperature is preferably between -15 °C and 120 °C, especially between 20 and 90 °C. Preferably rhombohedral, scalenohedron and spherical crystals are formed,

whereby the crystals measure between 0.05 and 5  $\mu\text{m}$ , especially between 0.3 and 2.5  $\mu\text{m}$ .

Static and/or moving, especially rotating mixing elements may be utilized for the production of a fibrous stock suspension which is loaded with calcium carbonate.

The process is preferably carried out in a pressure range of between 0 and 15 bar, especially between 0 and 6 bar. Also, the process is carried out at an pH value that is preferably between 6 and 10, especially between 6.5 and 9.5. The reaction time is here between 0.01 and 180 seconds, especially between 0.05 seconds and 60 seconds.

An additional advantage when utilizing the inventive technology with the above referenced paper grades consists in that these can also be further processed in a calendar. Due to the fact that fiber loading particles are deposited in, around and on the fibers, when utilizing the fiber loading technology blackening is avoided.

Fibrous stock produced with the fiber loading combination process technology possesses a superior dewatering characteristic when compared with a fibrous stock produced according to conventional methods; the improvement in the dewatering capacity is between 5 to 100 ml CSF or 0.2 to 15° SR, depending upon the required freeness and filler content. This fibrous stock possesses a low water retention value of 2 to 25%, depending upon the raw material that is used in production. Compared to conventional fibrous stock the water can be removed quicker from the fibrous stock suspension, and the fibrous stock dries accordingly faster. This also has a positive effect on remoistening which as a result has diminished in the paper production process, and upon the printability of the produced paper grades.

The current invention relates also to a device for the implementation one of the methods described previously. For this purpose a static mixer in which the fibrous stock suspension is washed is located prior to a dewatering screw.

An additional embodiment of the invention provides that fiber stock filtrate which was yielded in the dewatering screw can be returned via a pipe to a header tank or to another upstream device for the preparation of the fibrous stock suspension.

Preferably, an additional static mixer in which the fibrous stock suspension is washed is installed prior to a crystallizer.

Another advantageous provision is to locate an additional washer for cleaning of the fibrous stock suspension, following the crystallizer.

In another embodiment of the apparatus an additional static mixer in which the fibrous stock suspension is mixed with a filtrate and/or a calcium hydroxide suspension is located prior to the crystallizer.

The invention is described in further detail below, with the assistance of design examples and drawings:

Figure 1        a first schematic drawing – preparation of a fibrous stock suspension for application in a machine for the production of a fiber web and

Figure 2        a second schematic drawing.

A pipe line system 1 (Fig. 1) that is equipped with control valves 2, 3 is provided for a fibrous stock suspension. The control valve 2 is located in a pipe 4 through which the pipe line system 1 is connected with a static mixer 5. Dilution water and/or preferably filtrate that is interlaced with calcium hydroxide is added to the mixer 5 via a valve 6. A tank 7 or a container for storage of the fibrous stock suspension is located following the



mixer 5, viewed in direction of fiber stock flow direction. From the tank 7 the fibrous stock suspension is pumped via a pump 8 to an additional static mixer 9. Dilution water is also added to the mixer 9 via a valve 10. Likewise, the inflow of a calcium hydroxide suspension is controlled through a valve 11 which is located in a line 12.

This is supplied by a preparation unit 13 where solid calcium oxide or calcium hydroxide is added to water. For this purpose the preparation unit 13 is supplied via a line 14 which is equipped with a valve 15 with water. The suspension that is produced in the preparation unit 13 is directed through a pump 16 into a line 12.

The fibrous stock suspension to which calcium hydroxide was added flows from the mixer 9 into a line 17 through a valve 18 to a dewatering screw 19 where water is removed from the fibrous stock suspension. The water can, for example be returned through a line 20 to the mixer 5 as dilution water. Alternatively, or additionally the water that was removed in the dewatering screw 19 may also be routed to a storage tank 21 for the fibrous stock suspension, or it is returned to the mixer 9. Due to the return flow of calcium hydroxide containing water the pH value can be increased and adjusted, in all instances in the units that are located preceding the dewatering screw 19.

In order to equalize the fibrous stock suspension, said suspension is brought via a line 22 from the dewatering screw 19 to an equalizing screw 23. This is followed through a line 24 by a container 25 (crystallizer). For the purpose of supplying carbon dioxide this is connected with a carbon dioxide storage container 30 via a line 29 which is equipped with valves 26, 27 and a pump 28. Carbon dioxide is supplied from this container into the crystallizer 25 in order to produce the desired precipitation reaction of calcium hydroxide and carbon dioxide for the formation of calcium carbonate as a filler in the fibers of the fiber stock.

In addition the carbon dioxide container 30 is connected with the equalizing screw 23 via an additional line 31 which is equipped with a valve 32 and which branches off line 29. Carbon dioxide can herewith also be supplied to the equalizing screw in order to already achieve at least a partial precipitation there.

Line 29 is also connected via an additional valve 33 with a static mixer 34. This serves to add additional carbon dioxide to the fibrous stock suspension which is flowing from the crystallizer 25 via a line 36 which is equipped with a valve 35.

The fibrous stock suspension flows from the mixer 34 into a mixing chest 37. A filtration unit 38 may be provided between the mixer 34 and the mixing chest 37. From the filtration unit 38 the filtrate which has been enriched with calcium carbonate is returned into the header tank 7 or into an other downstream unit for the preparation of the dilution water or the fibrous stock suspension. The mixing chest 37 is equipped with a rotor 39 to thoroughly mix the fibrous stock suspension. From the mixer 34 the fibrous stock suspension then flows either immediately to a head box in a paper machine, or will be subjected to additional mechanical processing, for example in a refiner feed chest.

Fibrous stock suspension to which calcium hydroxide has not yet been added can be supplied to the mixer 34 through a pipe line system 1 via the valve 3 and a line 40 in which said valve is installed.

It is further provided that white water or process water which has for example been recovered in the wire area or the paper machine or, as already described previously, fibrous stock suspension from the dewatering screw 19 is supplied to the tank 21. Dilution water may for example be supplied to this tank through a line 41 which is equipped with a valve 42.

From the container 21 the dilution water which is mixed with process water flows through a line 43, a pump 44 and a valve 45 to the crystallizer 25. According to the design of an arrangement depicted in Fig. 1 for loading of the fibrous stock suspension with a filler, especially with calcium carbonate, a multitude of possibilities therefore arise to influence the composition of the fibrous stock suspension that is to be produced in various stages of the production process.

In another design form a fibrous stock suspension is transported for the purpose of being loaded with calcium carbonate in a device 48 (Fig 2) in a pipe line system that is equipped with control valves 49, 50. The control valve 49 is located in a line 51 through which the pipe line system is connected with a static mixer 52. This mixer can be supplied via a valve 53 with dilution water. Likewise, an additional valve 55 which is installed in line 54 controls the supply of a suspension of calcium hydroxide. This is supplied by a preparation unit 56 into which solid calcium oxide or calcium hydroxide is fed. For this purpose water is supplied into the preparation unit 56 via a line which is equipped with a valve 57. The suspension which is produced in the preparation unit 56 is brought into the line 55 via a pump 58.

Diluted suspension which has been treated with calcium hydroxide flows from the mixer 52 into a line 59 which is equipped with a valve 60. The suspension is fed immediately from the line 59 into a container 61 (Crystallizer). This is connected with a carbon dioxide storage tank 66 by means of a line 65 which is equipped with valves 62, 63 and with a pump 64 for the purpose of supplying carbon dioxide. Carbon dioxide is fed from said storage tank into the crystallizer 61 in order to create there the desired precipitation reaction of calcium hydroxide and carbon dioxide for the formation of calcium carbonate as a filler in the fibers of the fiber stock. Instead of utilizing a mixer 52 the calcium hydroxide can also be added from a header tank.

The line 65 is connected through an additional valve 67 with a static mixer 68 which serves to add additional carbon dioxide to the fibrous stock suspension flowing from the crystallizer 61 through a line 70 which is equipped with a valve 69.

From the mixer 68 the fibrous stock suspension flows into a blend chest 71 which is equipped with a rotor 72 for blending the fibrous stock suspension. From the blend chest 71 the fibrous stock suspension flows either immediately to a headbox in a paper machine or is subjected to additional mechanical processing, for example in a refiner feed chest.

The blend chest 71 can additionally be supplied via the valve 50 and a line 73 with fiber stock suspension which has not been treated with calcium hydroxide.

In addition, a refiner 74 may be incorporated in the pipe line system, serving the refining of the fiber stock suspension by means of an additional refining process. Said refiner is supplied with fiber stock suspension via a line 75 which branches off line 59. From the refiner 74 the fibrous stock suspension which has been refined again is brought through a line 76 into the line 70 and from there, as described above into the storage tank.

In addition, a provision can be made that carbon dioxide is supplied to the refiner 74 from the carbon dioxide storage tank 66 through a line 77 branching off line 65, and a static mixer 78 connecting line 77 with line 75.

In this arrangement for the preparation of the fibrous stock suspension the container 74 additionally assumes the refining process, thereby creating a simple design for the machine line for fiber stock preparation. The refining process serves at the same time as an agitation process in order to deposit the calcium carbonate in the fibers through a shear process.

A filtration unit 79, similar to the filtration unit 38 may be located also between the mixer 68 and the blend chest 71 from where filtrate treated with calcium carbonate is returned into a header tank or into another preceding unit for preparation of the dilution water or the fibrous stock suspension.